

PCT

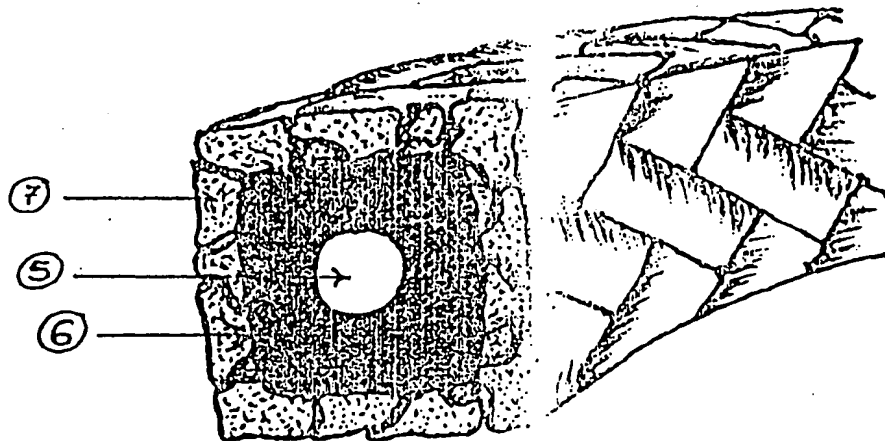
WORLD INTELLECTUAL PROPERTY ORGANIZATION  
International Bureau



INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(51) International Patent Classification <sup>6</sup> : D01D 5/42, F16J 15/22, D01F 6/12		A1	(11) International Publication Number: WO 99/60191
			(43) International Publication Date: 25 November 1999 (25.11.99)
(21) International Application Number: PCT/IN98/00001		(81) Designated States: US, European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE).	
(22) International Filing Date: 29 December 1998 (29.12.98)			
(30) Priority Data: 318/Bom/98 20 May 1998 (20.05.98) IN		Published With international search report. With amended claims and statement.	
(71)(72) Applicant and Inventor: PANDEY, Raj, Kumar [IN/IN]; Plot No. A/465, Road No. 28, Wagle Industrial Estate, Thane 400 604, Mumbai, Maharashtra (IN).			
(74) Agent: BHATNAGAR, Mahendra, Prasad; Lall Lahiri & Salhotra, N-128, Panchsheel Park, New Delhi 110 017 (IN).			

(54) Title: EXPANDED SINGLE MOLECULAR ALIGNED TEMPERATURE TREATED YARN



(57) Abstract

A high strength, expanded single molecular aligned temperature treated yarn for braiding purposes used in gland packing, wherein the PTFE extrusion grade paste or PTFE extrusion grade paste loaded with microfine graphite powder are mixed with odourless mineral spirit or oil or naphtha and put in a specialized container for loading of graphite into PTFE without shearing of PTFE particles by rotation, allowing the said mixture to age overnight to obtain a homogenous mixture, extruding the said mixture to obtain hollow tube of predetermined thickness, collapsing and slitting the said hollow tube to a fibrous mass, subjecting the said tape to heat treatment, stretching the said tape for molecular alignment of the fibers of the said tape, dipping the said molecular aligned tape in lubricating oil or mixture thereof, calendering the said lubricated tape through a set of rollers, passing the calendered tape through a temperature zone in an inert atmosphere, cooling the said tape to an ambient temperature and passing them through rollers covered with lubricants to obtain the uniform dimensions, high strength, yarn for braiding.

BEST AVAILABLE COPY

*FOR THE PURPOSES OF INFORMATION ONLY*

Codes used to identify States party to the PCT on the front pages of pamphlets publishing international applications under the PCT.

AL	Albania	ES	Spain	LS	Lesotho	SI	Slovenia
AM	Armenia	FI	Finland	LT	Lithuania	SK	Slovakia
AT	Austria	FR	France	LU	Luxembourg	SN	Senegal
AU	Australia	GA	Gabon	LV	Latvia	SZ	Swaziland
AZ	Azerbaijan	GB	United Kingdom	MC	Monaco	TD	Chad
BA	Bosnia and Herzegovina	GE	Georgia	MD	Republic of Moldova	TG	Togo
BB	Barbados	GH	Ghana	MG	Madagascar	TJ	Tajikistan
BE	Belgium	GN	Guinea	MK	The former Yugoslav Republic of Macedonia	TM	Turkmenistan
BF	Burkina Faso	GR	Greece	ML	Mali	TR	Turkey
BG	Bulgaria	HU	Hungary	MN	Mongolia	TT	Trinidad and Tobago
DJ	Dominica	IE	Ireland	MR	Mauritania	UA	Ukraine
BR	Brazil	IL	Israel	MW	Malawi	UG	Uganda
DY	Belarus	IS	Iceland	MX	Mexico	US	United States of America
CA	Canada	IT	Italy	NE	Niger	UZ	Uzbekistan
CF	Central African Republic	JP	Japan	NL	Netherlands	VN	Viet Nam
CG	Congo	KE	Kenya	NO	Norway	YU	Yugoslavia
CH	Switzerland	KG	Kyrgyzstan	NZ	New Zealand	ZW	Zimbabwe
CI	Côte d'Ivoire	KP	Democratic People's Republic of Korea	PL	Poland		
CM	Cameroon	KR	Republic of Korea	PT	Portugal		
CN	China	KZ	Kazakhstan	RO	Romania		
CU	Cuba	LC	Saint Lucia	RU	Russian Federation		
CZ	Czech Republic	LI	Liechtenstein	SD	Sudan		
DE	Germany	LK	Sri Lanka	SE	Sweden		
DK	Denmark	LR	Liberia	SG	Singapore		
EE	Estonia						

## EXPANDED SINGLE MOLECULAR ALIGNED TEMPERATURE TREATED YARN

The present invention relates to a high strength expanded single molecular aligned temperature treated yarn for braiding and filter elements having a better strength, performance, durability and life and process for the manufacture of the same.

Another object of the subject invention is that it relates to the use of these yarns in the manufacture of better performance gland packing and filter elements.

The other embodiment of the present invention relates to hollow core high recuperation packing for stern tube, rubber, stabilizer, pumps, mixers and agitators.

It has been found that the Tetra Fluoro Ethylene Polymer extruded in the form of a solid porous article resulted in poor strength. The conventional process comprises expanding a shaped article consisting essentially of higher crystalline poly tetra fluoroethylene made by a paste forming extrusion technique after removal of lubricant, by stretching at different rate. The cord extruded by this conventional method of making flat tape by calendering results in uneven expansion, due to compressing and shearing of the particle which are on the outer surface. The moment the particles on the outer surfaces are compressed and sheared, it results in the uneven expansion and dimensional variation in thickness and width of the tape.

The porous Tetra Fluoro Ethylene Polymer as claimed in U.S. Patent no.3,953,566 having amorphous content exceeding about 5% and having a micro structured characterized by nodes interconnected by fibrils though having high porosity and strength needs further strength and porosity to make the shaped article having better strength and long durability.

Though, the development of the invention of U.S. Patent No. 3,953,566 to Gore, flexible fibers made from expanded polytetrafluoroethylene (PTFE) have been used for a variety of purposes, including a fiber used as a thread and as a component in woven fabrics. The subject invention has resulted in the substantial improvement over previous materials.

Further, in U.S. Patent No. 5635,124 a process has been claimed for preparing a fibre comprising a strand of expanded polytetra fluoro ethylene by providing a expanded sheet of porous PTFE and slitting the sheet into multiple strands of fibres. However, the fibres so obtained proved to be lacking in compressibility, chemical inertness and tensile strength, which has been overcome in subject invention successfully.

The use of powder of PTFE was disclosed in U.S. Patent No. 5,591,526, where the fine powder of PTFE resin was blended with a lubricant, until a compound is formed. There the volume used was decided on the trial method as, should be sufficient to lubricate primary material of the PTFE resin to minimize the potential of the shearing of the particles prior to extruding. The process such used leads to certain disadvantages which was overcome in the subject invention when the paste of PTFE was used in specific amount and quantities.

The properties of the fibre made in accordance with the subject invention has proved to be considerably beneficial as compared from previous PTFE and expanded PTFE fibres. A conventional porous PTFE fibre such that sold under the trademark RASTEX by W.L.Gore & Associates have performed very well where porosity, fabric finish and thickness are not critical but the improved fibre of subject invention has overcome all these criticalities.

The objective of the present invention is accomplished by a process involving paste form product of a Tetra Fluoro Ethylene Polymer to make them both porous and stronger.

Though paste forming of dispersion polymerized poly(Tetra Fluoro Ethylene Polymer) is well known commercially. The steps in paste forming includes mixing the resin with a lubricant which is later removed by the process of drying. In usual practise the unsintered product is heated above the polymers melting point causing it to sinter or coalesce into an essentially impermeable structure. Such paste-formed, dried unsintered shapes could be expanded by stretching them in one or more

directions under certain conditions but they were not found to be of desired strength and porosity.

To overcome the above referenced problem and to attain high strength in the present invention, the PTFE paste extrusion grades are mixed with mineral oil or neptha or PTFE extrusion grade is loaded with microfine graphite powder and amorphous carbon in a special blender container where the blending or loading is carried out without shearing of particles. The container in which PTFE and Mineral Oil and or graphite and carbon are put in predetermined fixed quantity (for white grade no graphite and carbon) and for better lubricity yarn graphite and carbon in predetermined maximum percentage exceeding 50% are loaded in to the PTFE in this specialized container rotating at low RPM. The special blades on the sides of the container blends the mixture together uniformly with specially made bristles brush wiping them continuously without shearing of the particles. The PTFE extrusion grade- Teflon-GJ is mixed with MTO or Naptha and is loaded with microfine graphite powder and amorphous carbon and mixed together in predetermined quantity (for white grade no graphite and carbon) maximum percentage exceeding 50%.

Further, the stretching and the treatment steps are very vital steps which makes the product more versatile having better strength and durability. The main embodiment of the invention resides in the improved process for the manufacture of tapes having uniform dimension of thickness and width giving better strength and durability.

The embodiment of the invention resides in extracting yarns from the collapsed and silted hollow tubes by means of conventional yarns separating means, treating the individual tow and winding them to make a versatile, durable and high strength yarns by temperature stretching the slitted tape and then separating the yarns. The temperature stretch is again applied which is used for braiding purposes giving better strength and durability.

The increase in strength of the polymer matrix is dependent upon the strength of the extruded material before expansion, the degree of crystallinity of the polymer, the rate

and temperature at which the expansion is performed and amorphous locking. When all these factors are employed to maximize the strength of the material the high tensile strength and porosity is obtained.

The hollow tube of the subject invention extruded with a predetermined diameter and thickness, not only obviates the disadvantageous inherent in the conventional extruded solid cord but gives an extra strength due to the homogenous alignment of PTFE along with the periphery. To attain the optimum desired homogenous alignment it is essential that the wall thickness and diameter of the tube is predetermined keeping in view the ingredients, extrusion pressure. In the subject invention the optimum configuration is achieved by having a wall thickness of the extruded tube from 0.5mm to 5mm diameter and the diameter of the hollow core to be 1mm to 15mm.

To attain high strength in the present invention, the PTFE paste extrusion grade is mixed with mineral oil or neptha or PTFE extrusion grade is loaded with microfine graphite powder and amorphous carbon in a special blender container where the blending or loading is carried out without shearing of particles. The container in which PTFE and Mineral Oil and or graphite and carbon are put in predetermined fixed quantity (for white grade no graphite and carbon) and for better lubricity yarn graphite and carbon in predetermined maximum percentage exceeding 50%, are loaded in to the PTFE in this specialized container rotating at low RPM. The special blades on the sides of the container blends the mixture together uniformly with specially made brush having bristles wiping them continuously without shearing of the particles.

Further, due to continuous deforming and reforming the gland packing inside the stuffing box becomes loose and breaks down causing uncontrolled leakage. This occurs because in a reactor, stern, mixer or an agitator, usually the shaft size is very big and the length of the shaft is also very long, hence the slightest ovality in the shaft results in an excessive run out. This runout continuously deforms and reforms the gland packing in the stuffing box and results in the loosening and failure of such gland packing.

To overcome this problem and for the effective sealing , a gland packing should have an excellent rate of recuperation without the danger of braiding coming loose and breaking down.

The hollow core packing is designed keeping in view the very aspect of deformation and reformation.

The concept of the hollow core packing is designed and custom built for each application. The measurement of runout , type of fluid, gas required to be sealed against the temperature and pressure are the parameters required to design the core and the covering braid depending upon particular application. The hollow core takes care of the excessive runout without damaging the braid due to constant deforming and reforming as in the case of an ordinary packing.

Hollow core has to be designed keeping in view the following parameters:

- 1) Type of equipment
- 2) shaft size
- 3) Amount of runout of the shaft
- 4) Amount of scoring of the shaft
- 5) Packing size
- 6) Media i.e. the gland packing used to seal against chemicals and gases
- 7) Temperature
- 8) Pressure
- 9) Shaft speed

The rubber core used should have the required chemical resistance against the media and the temperature resistance. The round hole inside the square tube is designed keeping in view the amount of run out i.e. if the runout of the shaft is 6mm, the inner hole should be minimum 4 to 6mm thick. The rubber should have minimum of 40 to 80 shore hardness. Since, the minimum wall thickness required is 4 to 6mm.

The rubber core may be made of silicone, viton, ethylene, propylene, nitrile, neoprene, butyl rubber etc. or a combination of any of them best suited to resist the temperature and the chemicals inside the equipment.

The outer braiding can be either, the one which is disclosed above or the combination of Fluoro polymer, PTFE or Graphite/PTFE only or Fluoro polymer, PTFE or graphite/PTFE reinforced with kevlar fibre, carbon fibre, glass fibre or nylon fibre, the same have been disclosed in detail in the U.S. Patent application No. 09/100,066 dated June 19, 1998 corresponding to Indian Patent Application Number 276/BOM/97.

### **BRIEF DESCRIPTION OF THE DRAWINGS**

Figure 1 relates to the conventional packing showing the distribution of gland follower load

Figure 2 relates to the graph of conventional packing showing that the maximum pressure on the gland packing is near the gland follower

Figure 3 relates to the cross-section view of the developed hollow core high recuperation packing

Figure 4 relates to the uniform gland load distribution

### **DETAILED DESCRIPTION OF THE INVENTION:**

In the present invention, in order to get high strength, expanded single molecular aligned temperature treated yarn, the PTFE extrusion grade paste or PTFE extrusion grade paste loaded with microfine graphite powder of size 150-220 microns. The PTFE paste extrusion grade either alone or with graphite powder are put into a round transparent container and 10-40 % by weight mineral spirit or oil or neptha is added. The container in which PTFE and Mineral Oil and or graphite and carbon are put in predetermined fixed quantity (for white grade no graphite and carbon) and for better



lubricity yarn graphite and carbon in predetermined maximum percentage exceeding 50% are loaded in to the PTFE in the specialized container rotating at low RPM. The special blades on the sides of the container blends the mixture together uniformly with specially made brush having bristles wiping them continuously without shearing of the particles. Normally PTFE paste extrusion grade does not accept more than 15% loading of carbon. The extra loading of graphite for lubrisity is possible only due to this innovative specialized technique.

The said homogenous mixture is loaded in a hydraulic barrel for extrusion under a pressure of 20-500kgs. The extruder is provided with the proper die having a predetermined diameter for extruding a hollow tube having the desired diameter and wall thickness. The pre-determined diameter and wall thickness of hollow tube plays a very important and significant roll in determining the overall strength of the end product. The hollow tube thus obtained is then immediately collapsed and slited to the required size and wound on different bobbins. The slited and collapsed tube is further treated to a high temperature by feeding it through a temperature zone of 50-300°C where the stretching and expanding is carried out about 20% to 1000%. Stretching the said tape for single axis molecular aligned of the fibers of the said tape, dipping the said single axis molecular aligned tape in mineral oil/ light paraffin oil or heavily lubricating oil or mixture thereof. It is then calendered through a set of rollers to obtain the thickness of 0.5mm to 0.05mm. The calendered tape is further passed through a temperature zone of 370-400°C for a period of 5seconds -5minutes preferably in an inert atmosphere.

The treated tape are then cooled by passing through water at an ambient temperature and dried preferably in an open atmosphere. The dried tape or tow is further passed through rollers covered with lubricants such as micro fined graphites molybdenum disulphide and the like. The tapes thus softened has uniform dimension of thickness and width and is then used for braiding.

After the yarn of desired thickness is manufactured by the above process the same is braided on the hollow core for the gland packing.

Figure 1 shows the conventional gland packing where the shaft (2) is having the conventional gland packing (3). The load of the packing (3) is minimum while the load on the packing (4) is maximum which can be seen by compression (4). Such distribution of load has been shown in the graph as depicted in figure 2 which indicates that the load on gland packing (3) is just 10% while at the gland packing (4) it is maximum of 100% thus causing wear of shaft as well as packing itself.

The Figure 3 is depicting the cross-section view of the hollow core high recuperation packing which comprises an inner hollow square core (6) made of rubber having an inner hole (5). The size of the inner hole (5) is determined by the size of the packing to consider the required amount of run out or sag of shaft of the equipment. The inner hole (5) is generally 2mm to 24mm. The wall thickness of square hollow core made of rubber is 2.5mm to 20mm which should be minimum 15% to 20% or more the actual gland packing size. The rubber square hollow core is having the braiding of reinforced yarns (7) which is generally synthetic fibre enveloped with PTFE or Graphite/PTFE. The braiding (7) varies from 2mm to 6mm. This braiding is also dependent on the size of the packing.

Figure 4 depicts the uniform load distribution, which is not acquired by the conventional gland packing as shown in figure 2.

The above referenced application can better be understood with reference to the foregoing examples.

#### EXAMPLE 1

A process for the manufacture of a high strength, single molecular aligned temperature treated yarn for braiding and filter elements wherein:-

The PTFE extrusion grade paste mixed with 12-38% by weight with microfine graphite powder of size 155-200 microns. The PTFE extrusion grade paste with graphite powder are put into a round transparent container and 18-32% by weight

mineral spirit is added, rotating at low RPM of 22-58 RPM for  $3^{1/2}$ - $5^{3/4}$  hours and put in a specialized container for loading of graphite into PTFE without shearing of PTFE particles by rotating at 20-60 RPM for 3-6 hours. The said homogenous mixture is loaded in a hydraulic barrel for extrusion under a pressure of 25-480kgs. The extruder is provided with the proper die having a pre-determined diameter for extruding a hollow tube having the desired diameter and wall thickness. The hollow tube thus obtained is then immediately collapsed and slitted to the required size and wound on different bobbins. The slitted and collapsed tube is further treated to a high temperature by feeding it through a temperature zone of 55-305°C where the stretching and expanding is carried out about 30% to 1010%. Stretching the said tape for single axis molecular alignment of the fibers of the said tape, dipping the said single axis molecular aligned tape in mineral oil/ light paraffin oil or heavily lubricating oil or mixture thereof. It is then calendered through a set of rollers to obtain the thickness of 0.55mm to 0.006mm. The calendered tape is further passed through a temperature zone of 350-420°C for a period of 5 seconds -  $4^{1/2}$  minutes in an inert atmosphere.

The treated tape are then cooled by passing through water at an ambient temperature and dried in an open atmosphere. The dried tape or tow is further passed through rollers covered with lubricants such as micro fine graphites molybdenum disulphide. The tapes thus softened have uniform dimension of thickness and width and is then used for braiding.

#### EXAMPLE-2

A process for the manufacture of a high strength, single molecular aligned temperature treated yarn for braiding and filter elements wherein:-

The PTFE extrusion grade paste is put into a round transparent container and 18-32% by weight mineral oil is added, rotating at low RPM for  $3-4^{1/2}$  hours and put in a specialized container for loading of graphite into PTFE without shearing of PTFE particles by rotating at 25-60 RPM for 3-6 hours. The said homogenous mixture is loaded in a hydraulic barrel for extrusion under a pressure of 35-540kgs. The extruder is provided with the proper die having a pre-determined diameter for

extruding a hollow tube having the desired diameter and wall thickness. The hollow tube thus obtained is then immediately collapsed and slitted to the required size and wound on different bobbins. The slitted and collapsed tube is further treated to a high temperature by feeding it through a temperature zone of 60-295°C where the stretching and expanding is carried out about 30% to 1010%. Stretching the said tape for single axis molecular aligned of the fibers of the said tape, dipping the said single axis molecular aligned tape in mineral oil/ light paraffin oil or heavily lubricating oil or mixture thereof. It is then calendered through a set of rollers to obtain the thickness of 0.55mm to 0.006mm. The calendered tape is further passed through a temperature zone of 350-420°C for a period of 4 seconds -6 minutes in an inert atmosphere.

The treated tape are then cooled by passing through water at an ambient temperature and dried in an open atmosphere. The dried tape or tow is further passed through rollers covered with lubricants such as micro fined graphites molybdenum disulphide. The tapes thus softened has uniform dimension of thickness and width and is then used for braiding.

### EXAMPLE 3

A process for the manufacture of a high strength, single molecular aligned temperature treated yarn for braiding and filter elements wherein,

The PTFE extrusion grade paste is put into a round transparent container and 18-32% by weight neptha is added. rotating at low RPM of 25-60 RPM for 3-4<sup>1/2</sup> hours and put in a specialized container for loading of graphite into PTFE without shearing of PTFE particles by rotating at 20-60 RPM for 3-6 hours. The said homogenous mixture is loaded in a hydraulic barrel for extrusion under a pressure of 35-540kgs. The extruder is provided with the proper die having a pre-determined diameter for extruding a hollow tube having the desired diameter and wall thickness. The hollow tube thus obtained is then immediately collapsed and slitted to the fibrous mass, which may then be slitted into strands by slitting means.

After the process of slitting the strands are further treated to a high temperature by feeding it through a temperature zone of 60-295°C where the stretching and expanding is carried out about 30% to 1010%. Stretching the said strands and dipping the said single axis molecular aligned tape in mineral oil/ light paraffin oil or heavily lubricating oil or mixture thereof. It is then calendered through a set of rollers to obtain the thickness of 0.55mm to 0.006mm. The calendered tape is further passed through a temperature zone of 350-420°C for a period of 4 seconds -6 minutes in an inert atmosphere. Once the such sheets are formed they are further sent to a set of cutting means for expansion in the longitudinal directions.

The width of the fibre can be controlled by several process known in the art of expanding PTFE.

The properties of the fibre made in accordance with the above procedures differ considerably from previous PTFE and expanded PTFE fibres. A conventional porous PTFE fibre such that sold under the trademark RASTEX by W.L.Gore & Associates have performed very well where porosity, fabric finish and thickness are not critical but the improved fibre of subject invention has overcome all these criticalities.

The subject invention has proved to be more advantageous as the subject invention resulted in the manufacture of high strength of even dimension yarns for braiding and filter elements.

The inventive fibre has a smoother surface than the conventional fibres, which resulted very beneficial as having less chances of fibrillation and thus providing superior release properties when woven into a sheet.

It has resulted in having better strength, good performance, durability and longer life as compared to other conventional yarns.

Hence, the improved processing steps of the present invention create a fibre that has a number of improved properties, including more uniform dimensions along its length,

improved compressibility and handling along with easy high quality uniform processing.

The subject invention has provided the use of these yarns in the manufacture of better performance gland packing and filter elements as compared to commercially available conventional yarns.

While particular embodiments of the present invention has been illustrated and described herein, the present invention should not be limited to such illustrations and descriptions. It should be apparent that changes and modifications may be incorporated and embodied as part of the present invention within the scope of the claims.

## WE CLAIM:

1 A process for the manufacture of high strength, expanded single molecular aligned temperature treated yarn for braiding purposes used in gland packing wherein the PTFE extrusion grade paste or PTFE extrusion grade paste loaded with microfine graphite powder of size 150-220 microns are mixed with 10-40% by weight of odourless mineral spirit or oil or neptha and put in a specialized container for loading of graphite into PTFE without shearing of PTFE particles by rotating at 20-60 RPM for 3-6 hours, allowing the said mixture to age overnight to obtain a homogenous mixture, extruding the said mixture under a pressure of 20-500kgs/cm<sup>2</sup> to obtain hollow tube of predetermined thickness, collapsing and silting the said hollow tube to a fibrous mass, subjecting the said tape to heat treatment at 50-350 °C, stretching the said tape for molecular alignment of the fibers of the said tape, dipping the said molecular aligned tape in lubricating oil or mixture thereof, calendaring the said lubricated tape through a set of rollers, passing the calendared tape through a temperature zone of 300-400 °C in an inert atmosphere, cooling the said tape to an ambient temperature and passing them through rollers covered with lubricants to obtain the uniform dimensions, high strength, yarn for braiding.

2 A process for the manufacture of high strength, expanded single molecular aligned temperature treated yarn for braiding purposes used in gland packing, as claimed in claim 1, wherein the said mixing is carried out in a closed container at a low RPM 25-50, comprising the angular blades on the side of the container for blending the mixture uniformly with specially made brush having bristles wiping them continuously without shearing thus carrying out the blending.

3 A process for the manufacture of high strength, expanded single molecular aligned temperature treated yarn for braiding purposes used in gland packing, claimed in claim 1, wherein the said fibres (tow) are single molecular aligned temperature treated fibres.

- 4 A process for the manufacture of high strength, expanded single molecular aligned temperature treated yarn for braiding purposes used in gland packing, claimed in claim 3, wherein said fibres mass (tow) is separated from the said tape after the first heat treatment by means of fibre separating device at 100-200°C and further treatment and stretching at 100-350°C.
5. A process for the manufacture of high strength, expanded single molecular aligned temperature treated yarn for braiding purposes used in gland packing, claimed in claim 4, wherein the stretching is carried out at the rate of 20-1000% in a temperature zone of 50-350°C at a rate of 20-1000% per Sec to per minute.
6. A process for the manufacture of high strength, expanded single molecular aligned temperature treated yarn for braiding purposes used in gland packing, claimed in claim 1, wherein the said fibers are subjected to 300-400°C in an inert atmosphere and cooled to ambient temperature.
7. A high strength, expanded single molecular aligned temperature treated yarn for braiding purposes used in gland packing, as claimed in claims 1-6, wherein the said PTFE extrusion grade powder is mixed with neptha.
8. A braided gland packing and filter element using a high strength, expanded single molecular aligned temperature treated yarn for braiding purposes, as claimed in any of the preceding claims.
- 9 A high strength, expanded single molecular aligned temperature treated yarn for braiding purposes used in gland packing, comprising a hollow core high recuperation packing with excessive shaft run out wherein a inner hollow square core made up of rubber having a round inner hole and an outer braiding consisting a plurality of reinforced yarns of a high strength, expanded single molecular aligned temperature treated yarn made up of PTFE and graphite powder mixed with mineral spirit to make the homogenous mixture and finally treating the same with the lubricant oils.



10. A high strength, expanded single molecular aligned temperature treated yarn for braiding purposes used in gland packing as claimed in claim 11, wherein the said hollow core high recuperation gland packing, is having the said round inner hole of the said hollow square core varying from 2mm to 24mm.

11. A high strength, expanded single molecular aligned temperature treated yarn for braiding purposes used in gland packing, as claimed in claim 11, wherein the said hollow core high recuperation gland packing is used for stern tube, rudder, stabilizer, pumps mixers, agitators and the like to reduce the packing wear in rotating equipment.

12. A high strength, expanded single molecular aligned temperature treated yarn for braiding purposes used in gland packing as claimed in claim 13, wherein the said hollow core high recuperation gland packing, is having the wall thickness of the said hollow square core varying from 2.5mm to 20mm.

13. A high strength, expanded single molecular aligned temperature treated yarn for braiding purposes used in gland packing as claimed in claim 14, wherein the hollow core high recuperation gland packing is having said round inner hole of the said hollow square core conforming to the cross-section size of the compression packing.

14. A high strength, expanded single molecular aligned temperature treated yarn for braiding purposes used in gland packing, wherein the hollow core high recuperation gland packing, having the thickness of the said outer braiding varying from 2mm to 6mm.

15. A high strength, expanded single molecular aligned temperature treated yarn for braiding purposes used in gland packing, wherein the said hollow core high recuperation packing, the said reinforced yarn comprises combination of synthetic fibre enveloped with PTFE or PTFE loaded with graphite powder.

## AMENDED CLAIMS

[received by the International Bureau on 29 July 1999 (29.07.99);  
original claims 9-15 cancelled; original claims 1 and 8 amended;  
remaining claims unchanged (2 pages)]

1 [AMENDED] A process for the manufacture of high strength, expanded single molecular aligned temperature treated yarn for braiding purposes used in gland packing wherein the PTFE extrusion grade paste or PTFE extrusion grade paste loaded with microfine graphite powder of size 150-220 microns mixed with 10-40% by weight of odourless mineral spirit or oil or neptha, is loaded in a container such as herein described, rotating the said container at a speed of 20-60 RPM for 3-6 hours to obtain the homogenous mixture without shearing of PTFE particles, allowing the said mixture to age overnight, extruding the said aged mixture under a pressure of 20-500kgs/cm<sup>2</sup> to obtain hollow tube of predetermined thickness, collapsing and slitting the said hollow tube to a fibrous mass tape, subjecting the said tape to heat treatment at 50-350 °C, stretching the said tape for molecular alignment of the fibers of the said tape, dipping the said molecular aligned tape in lubricating oil or mixture thereof, calendering the said lubricated tape through a set of rollers, passing the calendered tape through a temperature zone of 300-400 °C in an inert atmosphere, cooling the said tape to the ambient temperature and passing them through rollers covered with lubricants to obtain the uniform dimensions, high strength yarn, for braiding.

2 A process for the manufacture of high strength, expanded single molecular aligned temperature treated yarn for braiding purposes used in gland packing, as claimed in claim 1, wherein the said mixing is carried out in a closed container at a low RPM 25-50, comprising the angular blades on the side of the container for blending the mixture uniformly with specially made brush having bristles wiping them continuously without shearing thus carrying out the blending.

3 A process for the manufacture of high strength, expanded single molecular aligned temperature treated yarn for braiding purposes used in gland packing, as

claimed in claim 1, wherein the said fibres (tow) are single molecular aligned temperature treated fibres.

4 A process for the manufacture of high strength, expanded single molecular aligned temperature treated yarn for braiding purposes used in gland packing, as claimed in claim 3, wherein said fibres mass (tow) is separated from the said tape after the first heat treatment by means of fibre separating device at 100-200°C and further treatment and stretching at 100-350°C.

5. A process for the manufacture of high strength, expanded single molecular aligned temperature treated yarn for braiding purposes used in gland packing, as claimed in claim 4, wherein the stretching is carried out at the rate of 20-1000% in a temperature zone of 50-350°C at a rate of 20-1000% per Sec to per minute.

6. A process for the manufacture of high strength, expanded single molecular aligned temperature treated yarn for braiding purposes used in gland packing, as claimed in claim 1, wherein the said fibers are subjected to 300-400°C in an inert atmosphere and cooled to an ambient temperature.

7. A high strength, expanded single molecular aligned temperature treated yarn for braiding purposes used in gland packing, as claimed in claims 1-6, wherein the said PTFE extrusion grade powder is mixed with neptha.

8 [AMENDED]A high strength, expanded single molecular aligned temperature treated yarn for braiding purposes used in gland packing, whenever manufactured by the process as claimed in any of the preceding claims.

STATEMENT UNDER ARTICLE 19

The invention relates to the process for the preparation of a high strength expanded single molecular aligned temperature treated yarn and the product thus manufactured.

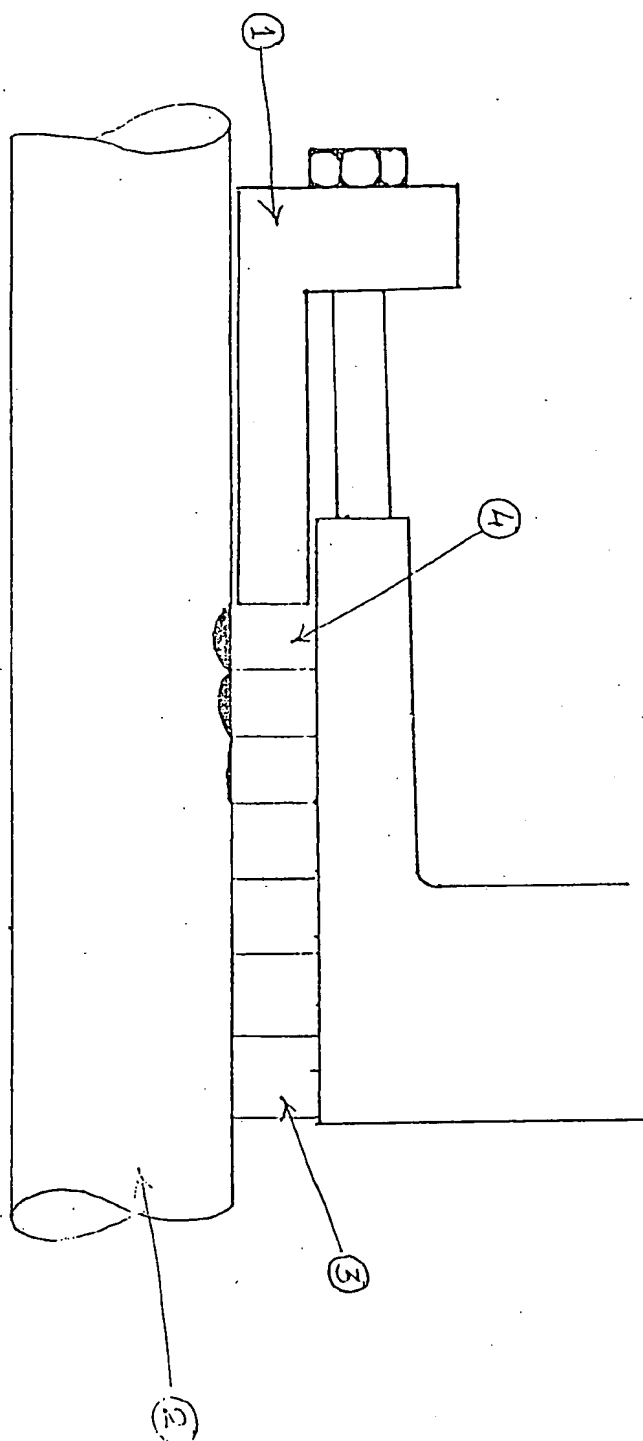
The novelty of the process and the product obtained by the process of subject application has got better tensile strength and porosity as the shearing of the PTFE particles is avoided which gives better fiber alignment and better fiber formation.

Further, the lubricants are not evaporated, thus giving a better extruded product having better porosity, strength and expansion.

In view of the amendemnt under Article 19(1), claims 1 to 8 are retained in the subject application, where minor re-phrasing have been conducted in the claims 1 and 8, to make the process of the invention more clear and explicit. The amendments so effectuated have been underlined in the amended claims.

Claims 9 to 15 are withdrawn from the subject application.

Fig. 1



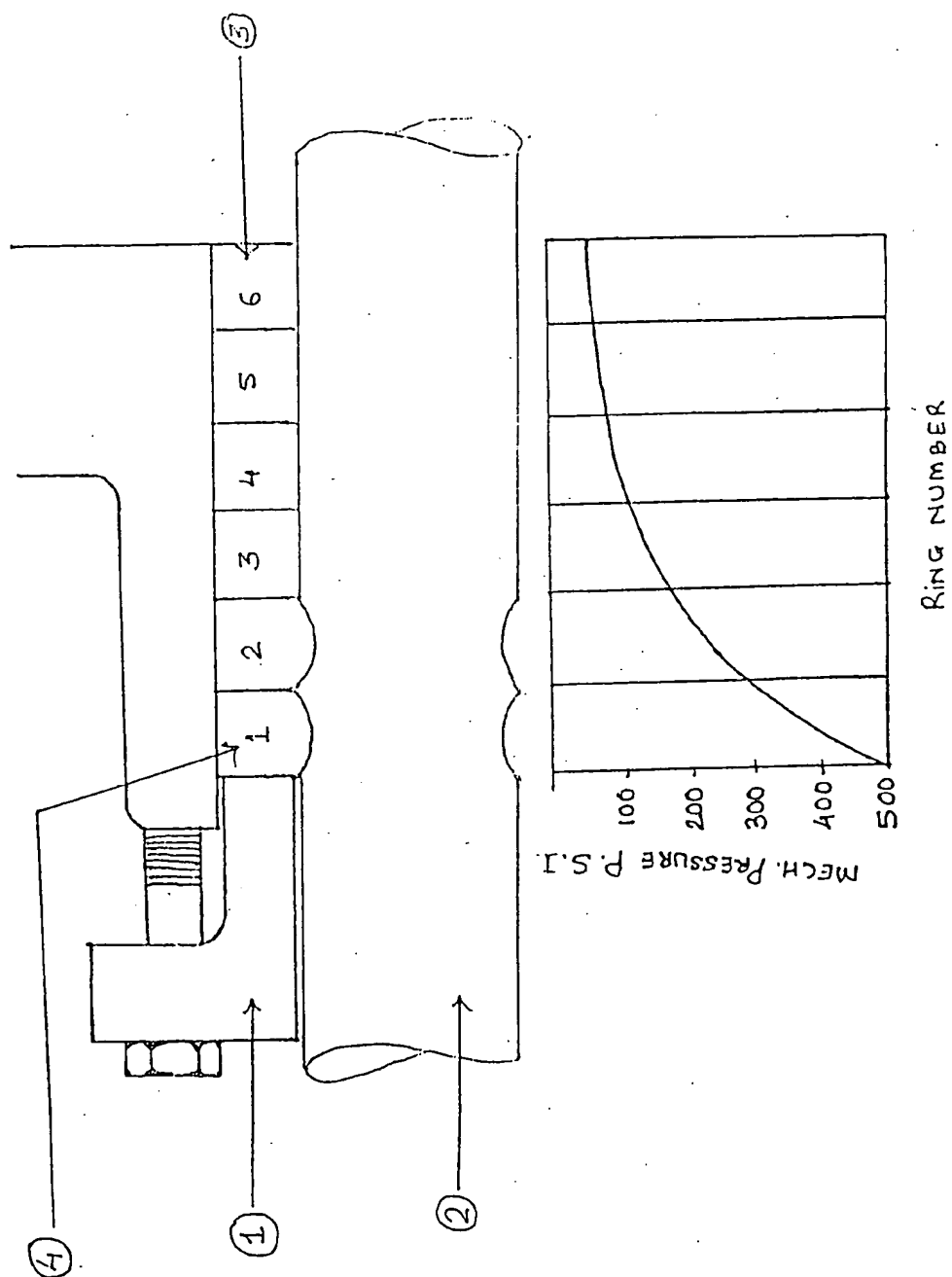


Fig. 2

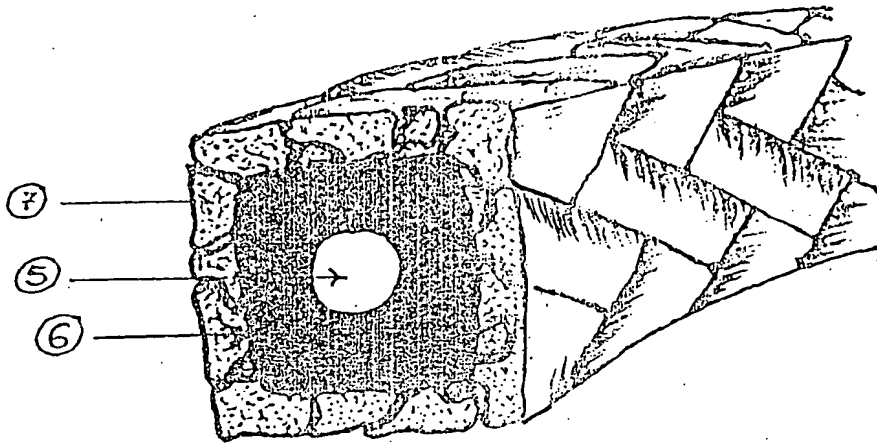


Fig. 3

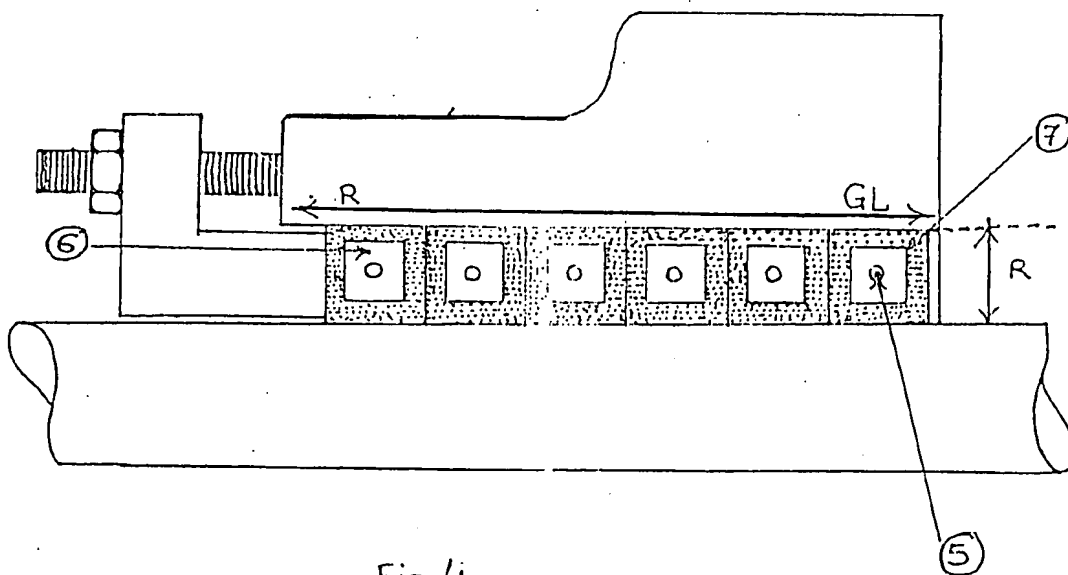


Fig. 4

# INTERNATIONAL SEARCH REPORT

International Application No.

PCT/IN 98/00001

A. CLASSIFICATION OF SUBJECT MATTER  
IPC 6 D01D5/42 F16J15/22 D01F6/12

According to International Patent Classification (IPC) or to both national classification and IPC

## B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC 6 D01D F16J D01F

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

## C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 3 953 566 A (GORE ROBERT W) 27 April 1976 cited in the application see column 1, line 58 - column 2, line 1 see column 3, line 66 - column 4, line 17 see column 5, line 11 - line 20 see column 10, line 15 - line 22; claims 1-5	1,3-8
X	EP 0 762 020 A (PROPACK DICHTUNGEN & PACKUNGEN) 12 March 1997 see column 1, line 10 - line 16 see column 2, line 19 - line 33; claims 1,2,5,6; figure 1	9-15



Further documents are listed in the continuation of box C.



Patent family members are listed in annex.

### \* Special categories of cited documents:

"A" document defining the general state of the art which is not considered to be of particular relevance

"E" earlier document but published on or after the international filing date

"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)

"O" document referring to an oral disclosure, use, exhibition or other means

"P" document published prior to the international filing date but later than the priority date claimed

"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

"X" document of particular relevance: the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

"Y" document of particular relevance: the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.

"&" document member of the same patent family

Date of the actual completion of the international search

14 May 1999

Date of mailing of the international search report

31/05/1999

Name and mailing address of the ISA

European Patent Office, P.B. 5818 Patentlaan 2  
NL - 2280 HV Rijswijk  
Tel. (+31-70) 340-2040, Tx. 31 651 epo nl,  
Fax: (+31-70) 340-3016

Authorized officer

Westermayer, W



# INTERNATIONAL SEARCH REPORT

International Application No  
PCT/IN 98/00001

## C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	<p>US 4 330 136 A (HENSON ROBERT W J)  18 May 1982  see column 1, line 54 - column 2, line 6;  figure 1</p> <p>-----</p>	9-15

# INTERNATIONAL SEARCH REPORT

information on patent family members

International Application No

PCT/IN 98/00001

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
US 3953566 A	27-04-1976	US 4096227 A	20-06-1978
		BE 767423 A	18-10-1971
		CA 962021 A	04-02-1975
		CH 555377 A	31-10-1974
		DE 2123316 A	02-12-1971
		DK 134342 B	25-10-1976
		FR 2090775 A	14-01-1972
		GB 1355373 A	05-06-1974
		JP 51018991 B	14-06-1976
		JP 1216843 C	17-07-1984
		JP 51030277 A	15-03-1976
		JP 56017216 B	21-04-1981
		JP 50138387 A	04-11-1975
		JP 55026567 B	14-07-1980
		NL 7107000 A,C	23-11-1971
		SE 392582 B	04-04-1977
		US 3962153 A	08-06-1976
		US 4187390 A	05-02-1980
		ZA 7103287 A	26-01-1972
EP 0762020 A	12-03-1997	DE 19532795 A	06-03-1997
US 4330136 A	18-05-1982	NONE	

**This Page is Inserted by IFW Indexing and Scanning  
Operations and is not part of the Official Record**

**BEST AVAILABLE IMAGES**

Defective images within this document are accurate representations of the original documents submitted by the applicant.

Defects in the images include but are not limited to the items checked:

- ☐ BLACK BORDERS
- ☐ IMAGE CUT OFF AT TOP, BOTTOM OR SIDES
- ☒ FADED TEXT OR DRAWING
- ☒ BLURRED OR ILLEGIBLE TEXT OR DRAWING
- ☐ SKEWED/SLANTED IMAGES
- ☐ COLOR OR BLACK AND WHITE PHOTOGRAPHS
- ☐ GRAY SCALE DOCUMENTS
- ☐ LINES OR MARKS ON ORIGINAL DOCUMENT
- ☐ REFERENCE(S) OR EXHIBIT(S) SUBMITTED ARE POOR QUALITY
- ☐ OTHER: \_\_\_\_\_

**IMAGES ARE BEST AVAILABLE COPY.**

**As rescanning these documents will not correct the image problems checked, please do not report these problems to the IFW Image Problem Mailbox.**